

Application No. 10/720,122
Declaration under 37 C.F.R. 1.132
Docket No.: 245926US0X DIV

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:
Toshio TSUJIMOTO, et al.

GROUP: 1792

SERIAL NO: 10/720,122

EXAMINER: Song, Matthew J.

FILED: November 25, 2003

FOR: SURFACE MODIFICATION PROCESS OF QUARTZ GLASS CRUCIBLE

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Yoshiyuki Tsuji who deposes and states that:

1. I am a graduate of Kyoto University and received my bachelor's degree in the year 1968.

2. I have been employed by Mitsubishi Materials Corp. for 16 years as a engineer in the field of quartz crucible.

3. The following calculations were carried out by me or under my direct supervision and control.

The thickness of the mixed metal oxide layer after baking of examples A-F as shown in Table 2 on page 18 in the specification was calculated as shown on the attached "Calculation method of thickness of mixed oxide layer." In this calculation, densities of the mixed oxide of SiO₂ and BaO are calculated based on a specific gravity of SiO₂ glass of 2.20 g/cm² and 5.72 g/cm² for BaO. As shown in the attached calculations, in Example A, the weight ratio of BaO/SiO₂ is 1/2 and therefore the volume of the metal oxide and the SiO₂ can be determined as 0.175 cm³ and 0.909 cm³ respectively. The total volume of Mixed oxide is therefore 1.084 cm³ and the density is calculated to be 2.77 g/cm³.

From Table 2, the measured adhesion amount of Metal oxide is $0.6 \mu\text{g}/\text{cm}^2$ and based on the ratio of BaO/SiO_2 of $1/2$, the total adhesion amount of Mixed Oxide is $1.8 \mu\text{g}/\text{cm}^2$ ($1.8 \times 10^{-6} \text{ g}/\text{cm}^2$).

The thickness of the Mixed oxide layer is equal to the Adhesion amount of Mixed Oxide/Density of the Mixed Oxide and as shown for Example A is $0.0065 \mu\text{m}$.

Correspondingly the values of Examples B through F are calculated and the results shown in the attached modified Table 2.

The calculated Mixed oxide layer thickness after baking according to the Examples of the claimed invention ranges from 0.0051 to $0.449 \mu\text{m}$. In comparison, in Example 1 of Watanabe (U.S. 6,106,610) the Ba-containing film has a thickness of about 0.2 mm ($200 \mu\text{m}$).

Therefore, the surface modified quartz glass crucible according to the claimed invention has an extremely thin layer of concentrated crystallization promoter. This layer is very effective to crystallize the inner surface of the crucible when the crucible is used for pulling up single crystal silicon at a temperature over 1400°C even though the coated layer is very thin. Therefore, the crucible as claimed in the above-identified application has a high resistance to deformation and a significantly reduced contamination contribution to the pulled up silicon single crystal. Thus the crucible of the claimed invention is superior in performance relative to conventional crucibles.

4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

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5. Further deponent saith not.

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Signature

Yoshiyuki Tsuji

Date

February 18, 2008

Calculation method of thickness of mixed oxide layer

Here, we use value of No.1, a kind of Silica Sol Liquid is A in Table 2 as an example.

1. Calculation of density of mixed oxide

Density of Mixed Oxide = Weight of Metal Oxide and SiO₂ / Volume of Mixed oxide

Volume of metal oxide = 1 / 5.72 = 0.175 cm³

Density of BaO: 5.72g/cm³ (Specific gravity of BaO)

Volume of SiO₂ = 2 / 2.2 = 0.909 cm³

Density of SiO₂: 2.2 g/cm³ (Specific gravity of SiO₂ glass)

Volume of Mixed Oxide = 0.175 + 0.909 = 1.084 cm³

Density of Mixed Oxide = 3 / 1.084 = 2.77 g/cm³

2. Calculation of adhesion amount of Mixed Oxide

Adhesion amount of Mixed Oxide = Adhesion amount of Metal Oxide + Adhesion amount of SiO₂

Adhesion amount of Metal Oxide = 0.6 μg/cm² (Measured Value in Table 2)

Adhesion amount of SiO₂ = 0.6 x 2 = 1.2 μg/cm²

BaO : SiO₂ = 1 : 2 (Weight Ratio in Table 2)

Adhesion amount of Mixed Oxide = 0.6 + 1.2 = 1.8 μg/cm² = 1.8 x 10⁻⁶ g/cm²

3. Calculation of thickness of Mixed Oxide layer

Thickness of Mixed Oxide layer = Adhesion amount of Mixed Oxide / Density of Mixed Oxide

Adhesion amount of Mixed Oxide = 1.8 x 10⁻⁶ g/cm²

Density of Mixed Oxide = 2.768 g/cm³

Thickness of Mixed Oxide layer = 1.8 x 10⁻⁶ g/cm² / 2.77g/cm³ = 6.5 x 10⁻⁷ cm = 6.5 x 10⁻³ μm (0.0065 μm)

The similar calculations were carried out to other examples in Table 2 so as to obtain MODIFIED TABLE 2.

MODIFIED TABLE 2

Silica Sol Liquid											
No.	Kinds	Oligomer solutions containing	Metal Salt	Dilution	Alchole	BaO Amounts (wt%)	SiO ₂ Amount (wt%)	Calculated Mixed Oxide density (g/cm ³)*	Adhesion Amount of Metal Oxide after Baking (μg/cm ²)	Calculated Adhesion Amount of Mixed Oxide after Baking (μg/cm ²)	Calculated Mixed Oxide Layer Thickness after Baking (μm)
1	A	(I) 20g	5wt.%	Calculated as BaO	20g	1	2	2.77	0.6	1.8	0.0065
2	B	(I) 20g	5wt.%	Calculated as BaO	40g	2	2	3.17	0.8	1.6	0.0051
3	C	(I) 5g	10wt.%	Calculated as BaO	50g	5	5	3.17	1	2	0.0063
4	D	(II) 40g	10wt.%	Calculated as BaO	50g	5	10	2.76	5.2	15.6	0.0565
5	E	(II) 80g	10wt.%	Calculated as BaO	20g	2	20	2.33	9.5	104.5	0.449
6	F	(I) 100g	Carbonic Acid Ba Powder	0.5g	-	0.4	10	2.26	2.1	54.6	0.242
7	G	Non-Surface Treatment							0		
8	H	Conventional Ba carbonate Powder							1		

* : Using densities are 5.72 g/cm³ for BaO and 2.2 g/cm³ for SiO₂.